Ultimate Mechanical Properties of Basalt Filaments

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ABSTRACT

Basic characteristics of basalt filaments are described. The main aim of this study is to explore tensile strength distribution based on experimental data. To select the appropriate failure risk function, the maximum likelihood method and methods based on order statistics are used. The use of basalt filaments in sewing thread constructions is also discussed.

Basalt Fibers

Basalt is a generic name for solidified lava from volcanos. Basalt rocks melt in the range of 1500 to 1700°C, and when the melt is quickly quenched, it solidifies to a nearly amorphous glass. Slow cooling leads to more or less complete crystallization into an assembly of minerals. Two essential minerals, plagiocene and pyroxene, make up perhaps 80% of many basalts [6]. From a chemical composition point of view, silicon dioxide dominates, with Al_2O_3 next in abundance, followed by CaO, MgO, and FeO. Basalt rocks are classified according to their SiO_2 content as alkaline basalts (up to 42% SiO_2), mildly acid basalts (43 to 46% SiO_2), and acid basalts (over 46% SiO_2). The color of basalt ranges from brown to a dull green depending on ferrous oxide (FeO) content. Basalts are more stable in strong alkalies than glass, but stability in strong acids is slightly lower. Basalt products can be used over a wide temperature, from about −200°C to about +800°C. At higher temperatures, structural changes begin to occur. For fiber preparation, basalt rocks must satisfy the following requirements: SiO_2 content above 46% (acid-type basalt) with constant composition, ability to melt without solid residue, appropriate melt viscosity for fiber formation, and ability to solidify to a glassy state without marked crystallization.

The main problems with uniform basalt fiber preparation are due to gradual crystallization of some mineral components in the melt (plagioclase, magnetite, pyroxene) and to nonhomogeneity of the melt. Basalt is therefore used mainly for molded products, such as flagstones and pipes, and in the form of short fibers (basalt wool) for insulation purposes. Continuous filament spinning overcomes the problems of unevenness, making the yarns suitable for producing planar or three-dimensional reinforcements in composites, special knitted fabrics, and sewing threads. Basalt sewing threads can be used for joining filter bags to use in agressive chemical environments at high temperatures.

Experimental

Basalt rocks from Vestany Hill (Czech Republic) were the raw material for filament yarn preparation. Table I lists the chemical composition of basalt filaments and typical glasses, and some basic physical characteristics are given in Table II.

TABLE I. Composition of glass and basalt fibers (in weight %).

Compound	E-glass	S-glass	C-glass	Basalt
SiO_2	52–56	65	64–68	51.56
Al_2O_3	12–16	25	3–5	18.24
CaO	16–25	-	11–15	5.15
MgO	0–5	10	2–4	1.30
B_2O_3	5–10	—	4–6	-
Na_2O	0.8	0.3	7–10	6.36
K_2O	0.8	0.3	7–10	4.50
TiO_2	-	-	-	1.23
Fe_2O_3	-	-	-	4.02
FeO	-	-	-	2.14

TABLE II. Basic physical characteristics of glass and basalt fibers.

Property	E-glass	Basalt
Diameter, μm	9–13	10.25
Density, kg m^{-3}	2540	2733
Softening temperature, °C	840	960
Stress at break, GPa	3.4 ± 0.7	1.43 ± 0.59

We measured the loads at break for individual basalt filaments under standard conditions on an Instron machine with digital output to an ASCII file. Load data were transformed to stresses at break σ_i (GPa). We used

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a sample set of 49 individual load-at-break values for statistical evaluation.

We also tested basalt filament yarns as sewing threads to join glass fabrics. During these tests, the basalt threads broke frequently due to their brittleness, so they were coated with poly(ethylene terephthalate) (PET) to form a composite thread [6]. The properties of these composite threads in comparison with glass sewing thread (TYGAFLOR) are shown in Table III.

TABLE III. Properties of glass and coated basalt sewing threads.

Property	TYGAFLOR	Basalt/PET
Fineness, tex	284 ± 2	283.3 ± 1.6
Thread diameter, mm	0.56 ± 0.02	0.72 ± 0.05
Strength at break, N tex^{-1}	0.32	0.34
Strength at break in loop, %	49.81	33.35
Elongation at break, %	1.87 ± 0.16	2.3 ± 0.2
Abrasion resistance, cycles	1214 ± 178	180 ± 49.8
Number of breaks in sewing test	8	0

Statistical Analysis of Fiber Strength

Fiber fracture can generally be described by micromechanical models or on the basis of purely probabilistic concepts. The probabilistic approach is based on the following assumptions: a fiber breaks at a specific place that has a critical defect (catastrophic flaw), defects are distributed randomly along the fiber length (Poisson model), and fracture probabilities at individual places are mutually independent.

The cumulative probability of nonfracture $C(V, \sigma)$ depends on the tensile stress level σ and the fiber volume V. A simple derivation of the stress at break distribution, described for example by Kittl and Diaz [2], leads to the general expression

$$C(V, \sigma) = \exp[-R(\sigma)] \quad , \tag{1}$$

where $R(\sigma)$ is the specific risk function.

The cumulative probability of break $F(\sigma)$ is a complement to $C(V, \sigma)$. The distribution of stress at break is then expressed as

$$F(\sigma) = 1 - \exp[-R(\sigma)] \quad . \tag{2}$$

For the Weibull distribution (model WEI3), $R(\sigma)$ has the form

$$R(\sigma) = [(\sigma - A)/B]^C \quad . \tag{3}$$

Here A is the lower strength limit, B is a scale parameter, and C is a shape parameter. For brittle materials, it is often assumed that $A = 0$ (model WEI2).

Weibull models are physically incorrect due to an unsatisfactory upper limit of strength (see Equation 2). To overcome this limitation, Kies [1] proposed a more general risk function (model KIES) in the form

$$R(\sigma) = [(\sigma - A)/(A_1 - \sigma)]^C \quad . \tag{4}$$

Here A_1 is the upper strength limit. For brittle materials, it is again assumed that $A = 0$ (model KIES2).

Generalization of the Kies risk function has been proposed by Phani [7] (model PHA5):

$$R(\sigma) = \frac{[(\sigma - A)/B_1]^D}{[(A_1 - \sigma)/B]^C} \quad . \tag{5}$$

In this equation, C and D are two shape parameters. We can prove that B and B_1 cannot be independently estimated. Therefore, the constraint $B_1 = 1$ is used in the following. A simplified version of Equation 4 has $A = 0$ (model PHA4).

For the well-known Gumbell distribution (GUMB), $R(\sigma)$ is expressed as

$$R(\sigma) = \exp[(\sigma - A)/B] \quad . \tag{6}$$

The selection of the right $R(\sigma)$ depends critically on the estimated number of modes and the presence or absence of a nonzero lower limiting strength.

ESTIMATING $R(\sigma)$ AND PARAMETERS

The objective of a statistical analysis of fiber strength data, $\sigma_i\, i = 1, \ldots N$, is the specification of a risk function $R(\sigma)$ and an estimation of its related parameters. Due to their special structure, the parameters of Weibull-type distributions can be estimated either by the maximum likelihood method or by quantile-based methods. Sometimes it is efficient to combine these and other methods to simplify the estimation process.

Maximum Likelihood Method (MLE)

The MLE method is interesting because of its good statistical properties (asymptotic efficiency, consistency, and asymptotic normality of estimators) [3]. For the case when $\sigma_i\ i = 1, \ldots N$ are independent random variables with the same probability density function $F'(\sigma_i, \mathbf{a})$, the logarithm of the likelihood function has the form

$$\ln L(a) = \sum_{i=1}^{N} \ln [F'(\sigma_i, \mathbf{a})] \quad , \tag{7}$$

where **a** are parameters of corresponding risk function.

The MLE estimators **a*** can be obtained by maximizing $\ln L(\mathbf{a})$. This task can be simply converted to solving the set of nonlinear equations (see reference 3). Estimates of **a*** obtained this way for three- and two-parameter Weibull distributions are given in Table

IV. The nonparametric quantile function estimator (Rosemblatt type, see reference 3) and WEI3 quantile function with parameters from Table IV are shown in Figure 1.

TABLE IV. Parameters of Weibull models calculated by MLE.

Model	A, GPa	B, GPa	C	$\ln L(\mathbf{a}^*)$
WE13	0.5327	0.431	6.5470	59.51
WE12	–	0.965	15.975	57.61

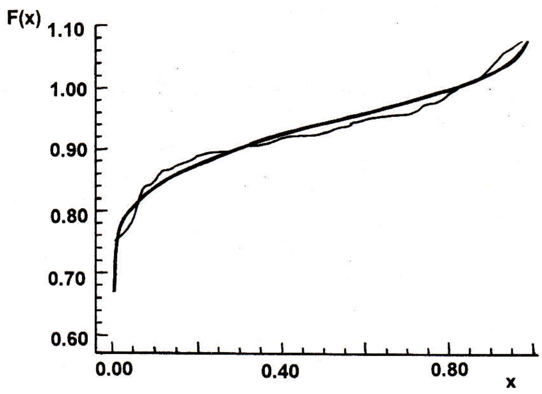

FIGURE 1. Nonparametric estimation of quantile function and WEI3 quantile function with parameter from Equation 1—thick line.

Quantile Based Methods

Methods of this sort use so-called order statistics $\sigma_{(i)}$, noting that $\sigma_{(i)} \leq \sigma_{(i+1)}$ $i = 1, \ldots N - 1$. It is well known that σ_i values are rough estimates of a sample quantile function for probabilities

$$P_i = F(\sigma_{(i)}) = \frac{i - 0.5}{N + 0.25} \quad . \tag{8}$$

By using Equation 16 and order statistics $\sigma_{(i)}$, the parameter estimation problem can be converted to a nonlinear regression task.

The so-called *Weibull transformation* method uses the rearrangement of Equation 9 for order statistics:

$$\ln [R(\sigma_{(i)})] = \ln [-\ln (1 - P_i)] \quad . \tag{9}$$

The parameter estimates of the $R(\sigma)$ model can then be obtained by nonlinear least squares, *i.e.*, by minimizing the criterion:

$$S(\mathbf{a}) = \sum_{i=1}^{N} [y_i - \ln R(\sigma_{(i)})]^2 \quad , \tag{10}$$

where $y_i = \ln [-\ln (1 - P_i)]$. Note that a graph of y_i versus $\ln (\sigma_{(i)})$ is a so-called Weibull plot. This plot for a two-parameter Weibul distribution is a straight line, but for a three-parameter distribution, a concave curve results. Parameter estimates obtained by minimizing Equation 10 are given in Table V. The regression curve for model WEI3 is shown in Figure 2.

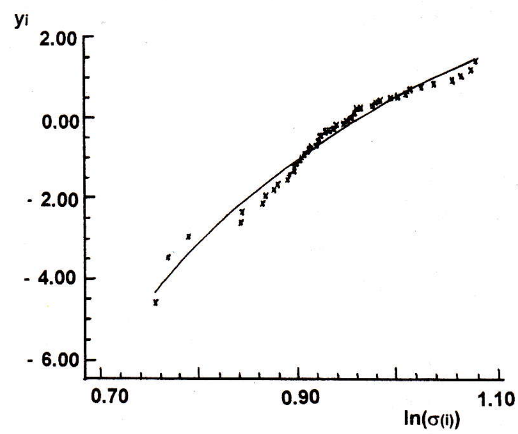

FIGURE 2. Weibull transformation regression for model WEI3.

Strictly speaking, the proposed method is based on the incorrect assumption that the y_i are uncorrelated random variables with constant variance. It is more logical to use the estimated sample quantiles $\sigma_{(i)}$ as explained quantities. A corresponding least squares criterion for the *quantile regression* has the form

$$S(\mathbf{a}) = \sum_{i=1}^{N} [\sigma_{(i)} - Q(Z_i)]^2 \quad , \tag{11}$$

TABLE V. Parameters of $R(\sigma)$ models calculated by regression in Weibull transformation.

Model	A, GPa	B, GPa	C	A_1, GPa	B_1 GPa	D	AIC	MEP
WEI3	0.548	0.479	6.19	–	–	–	−146.0	0.0596
WEI2	–	0.966	15.75	–	–	–	−132.8	0.0713
KIES	0.698	–	2.00	1.24	–	–	−128.0	0.0865
KIES2	–	–	8.15	1.94	–	–	−116.8	0.0996
GUMB	0.968	0.059	–	–	–	–	−115.5	0.1020
PHA4	—	1	21.59	1.13	0.077	−11.12	−157.9	0.0506
PHA5	−0.141	1	38.86	1.70	0.953	−15.10	−159.0	0.0480

where $Z_i = \exp(y_i)$ and $Q(Z_i)$ is a theoretical quantile function. For a three-parameter Weibull distribution, $Q(Z_i)$ is expressed as

$$Q(Z_i) = A + BZ_i^{1/C} \quad . \tag{12}$$

When a three-parameter Kies model is valid,

$$Q(Z_i) = \frac{A + A_1 Z_i^{1/C}}{1 + Z_i^{1/C}} \quad , \tag{13}$$

and for a Gumbell distribution,

$$Q(Z_i) = A + B \ln (Z_i) \quad . \tag{14}$$

According to the roughness of $\sigma_{(i)}$ and their nonconstant variances, we can define the special weights [2]. Parameter estimates obtained by minimizing Equation 11 are given in Table VI. The regression curve for model WEI3 is shown in Figure 3.

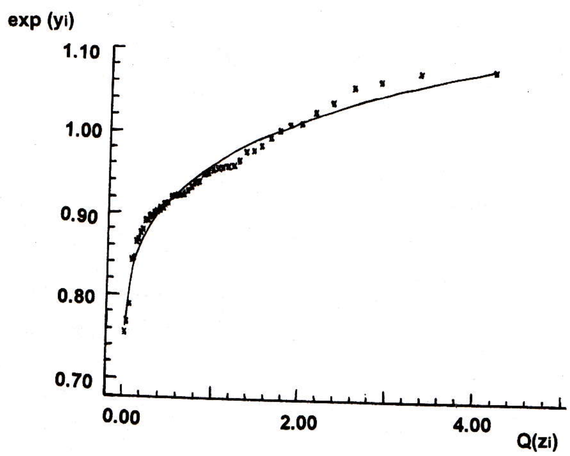

FIGURE 3. Regression curve for model WEI3.

Selecting an $R(\sigma)$ Model

Goodness-of-fit tests for comparing a sample distribution with a theoretical one with estimated parameters are often used [3]. Transforming parameter estimation problems in $R(\sigma)$ models to a regression problem enables us to use statistical criteria for selecting the optimal model form [4].

To distinguish between models with various numbers of parameters M, the Akaike information criterion (AIC) is suitable:

$$AIC = N \ln \left[\frac{S(\mathbf{a}^*)}{N - M}\right] + 2M \quad , \tag{15}$$

where $S(\mathbf{a}^*)$ is the minimal value of $S(\mathbf{a})$. The best model is considered to be that for which this criterion reaches a minimum.

The predictive ability of regression models can be examined using the mean quadratic error of prediction:

$$MEP = \frac{1}{N} \sum_{i=1}^{N} [y_i - f(x_i, \mathbf{a}_{-i})]^2 \quad , \tag{16}$$

where $f(x_i, \mathbf{a})$ is a model function. Parameters $\mathbf{a}_{-i}$ are least squares estimates when all points except the ith one are used. Criterion MEP is equal to the mean of the squared predictive residuals [4]. The best model with maximum predictive ability reaches a minimum of MEP. The AIC and MEP for individual models are given in Tables V and VI.

Discussion

An SEM micrograph of a typical broken basalt fiber (magnification 10,000) is shown in Figure 4. Evidently brittle fracture has occurred. An SEM of a longitudinal portion of basalt fiber (magnification 10,000) is shown in Figure 5. The surface is smooth and without flaws or crazes. Based on these findings, we can postulate that fracture occurs due to nonhomogeneities in the fiber bulk (probably near small crystallites of minerals).

The maximum likelihood method gives nearly the same results as the method based on regression in the Weibull transformation. Quantile regression gives more distinct estimates. The model proposed by Phani (Equation 13) leads to the parameter A without a physical sense. Model PHA4 is more realistic, but the shape estimates are very high. The Kies-type models (Equation 11) are no better that the three-parameter Weibull model, so the three-parameter Weibull model can be accepted as providing a suitable risk function.

The quantile-based methods are generally approximate, but the parameter estimation process is not as

TABLE VI. Parameters of $R(\sigma)$ models calculated by quantile regression.

Model	A, GPa	B, GPa	C	A_1, GPa	AIC	MEP
WEI3	0.601	0.031	4.32	–	−432.07	1.60×10^{-4}
WEI2	–	0.966	15.98	–	−410.70	2.40×10^{-4}
KIES	0.709	–	1.99	1.22	−407.90	2.45×10^{-4}
KIES2	–	–	8.95	1.93	−397.50	3.22×10^{-4}
GUMB	0.966	0.056	–	–	−395.80	3.37×10^{-4}

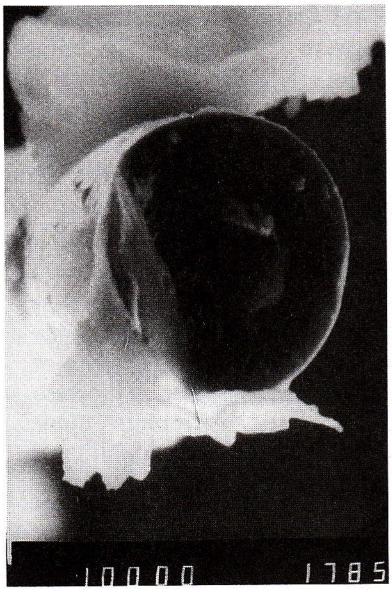

FIGURE 4. Broken end of basalt fiber (10,000×).

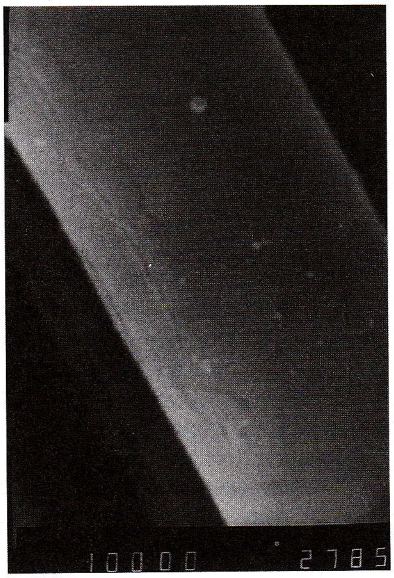

FIGURE 5. Longitudinal section of basalt fiber (10,000×).

difficult in comparison with the maximum likelihood method (see, *e.g.*, reference 3).

Conclusions

Basalt filaments are materials with great potential. For textile purposes, coating these filaments with polyester resin is necessary to improve their mechanical behavior. Brittle fracture is probably due to bulk defects and can be described by a three-parameter Weibull distribution. For parameter estimation, methods based on quantiles are attractive. A rough distinction between two- and three-parameter Weibull distributions can be made using moment-type estimators.

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